EVALUATION OF COAL-MINERAL ASSOCIATION AND COAL CLEANABILITY BY USING SEM-BASED AUTOMATED IMAGE ANALYSIS

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ABSTRACT

A technique employing SEM-based automated image analysis (AIA) has been developed for assessing the association of mineral particles with coal, and thus the cleanability of that coal, when the characteristics of the separation process are known. Data resulting from AIA include the mineral distribution by particle size, mineral phase, and extent of association with coal. This AIA technique was applied to samples of -325 mesh (-44 μm) coal from the Indiana No. 3, Upper Freeport, and Sunnyside (UT) seams. The coals were subjected to cleaning by float-sink separations at 1.3, 1.4, 1.6, and 1.9 specific gravity and by froth flotation. For the three coals, the float-sink procedure at a given specific gravity produced different amounts of clean coal, but with similar ash content. Froth flotation removed much less ash, yielding a product ash content of ~8% for the Upper Freeport coal, regardless of recovery, while reducing the ash content to less than 5% for the other two coals. The AIA results documented significantly more association of minerals with the Upper Freeport coal, which thus led to the poor ash reduction.

INTRODUCTION

The task of removing mineral matter from coal is greatly aided by a thorough characterization of the distribution and/or association of mineral and coal particles. Conventional characterization, based on laboratory float-sink and froth flotation tests at different sizes and conditions, is cumbersome and does not generally lead to any clear understanding of coal-mineral matter associations.

SEM-based automated image analysis (AIA) has unique capabilities for analyzing mineral particles in-situ for the important characteristics of size, phase, and association with the coal matrix. These AIA techniques have previously been applied to the determination of mineral matter size and phase distributions in coal (1-3) and are now being extended to the measurement of association. This work describes our application of AIA to characterize the association of minerals with coal and to correlate the AIA results with those of conventional tests such as float-sink separation and froth flotation.

EXPERIMENTAL

Coals

The characteristics of the three coals used in this study are presented in Table 1. The coals had been precleaned and ground to pass a 325 mesh screen. Since the conventional analyses give values for ash, whereas AIA provides results in terms of mineral matter, low temperature ash values were also obtained for easier comparison with AIA measurements.

Table 1. Characteristics of the three coals used (results are expressed as % of dry coal)

	Indiana No.3	Upper Freeport	Sunnyside
Proximate Analysis			
Ash	7.35	9.88	5.19
Volatile Matter	40.67	26.02	38.54
Fixed Carbon	51.98	64.10	56.19
Forms of Sulfur			
Total	4.26	1.56	0.61
Pyrite	2.23	0.95	0.05
Sulfate	0.07	0.01	0.00
Low Temperature Ash	10.5	12.5	6.1

Cleaning tests at AMAX

The coals were subjected to float-sink separations at the specific gravities (sp. gr.) of 1.3, 1.4, 1.6, and 1.9, and samples from each incremental sp. gr. range were weighed and analyzed. Another set of samples was subjected to froth flotation in an automated Denver batch cell, using 28 ppm MIBC and 0.54-1.92 ml kerosene per kg of coal. Froth was collected after 1.5, 3, 6, 12, and 24 minutes, and the remaining unfloatable material (tailings) was also collected for analysis.

SEM-based image analyses at Ames Lab

Samples of coal were embedded in carnauba wax, and a cross section was prepared for analysis as described previously (4). The AIA system included a JEOL JSM-U3 electron microscope, a Tracor-Northern TN-2000 energy-dispersive x-ray analyzer, and a LeMont Scientific DB-10 image analyzer. Samples were examined at magnifications of 300-400 diameters using an accelerating voltage of 25 kV and a beam current of 1-2 nA.

Software from LeMont Scientific was used to distinguish coal and mineral features from the background and from each other based on the brightness of their backscattered electron signal. Area and perimeter were measured for each feature, along with the fraction of perimeter in contact with each of the adjoining phases. Mineral particles were identified from the relative intensities of the characteristic x-ray emissions. X-rays were collected for 4 seconds per particle at a nominal rate of 500 counts per second. Data for contiguous features (i.e., with shared boundaries between coal and mineral matter) were grouped together so that the characteristics of the composite features could be determined.

RESULTS AND DISCUSSION

Conventional cleaning studies

The results of the float-sink separations are presented in Table 2. Data are presented for both the incremental fractions and for the cumulative fraction lighter than the indicated specific gravity. Plots of recovery and cumulative ash content are shown in Figure 1.

Table 2. Float-sink results for three coals (results are expressed as % of dry coal)

Sp. Gr.		Incremental			Cumulative		
Sink	Float	Weight	Ash	S(tot)	Weight	Ash	S(tot)
		Indiana No. 3 Coal					
	1.30	24.0	1.09	2.31	24.0	1.09	2.31
1.30	1.40	64.5	3.23	2.45	88.5	2.65	2.41
1.40	1.60	4.7	15.86	4.52	93.2	3.32	2.52
1.60	1.90	1.5	29.14	7.34	94.7	3.72	2.59
1.90		5.3	64.32	32.47	100.0	6.95	4.18
		Upper Freeport Coal					
	1.30	7.1	0.98	0.71	7.1	0.98	0.71
1.30	1.40	70.1	2.60	0.75	77.2	2.45	0.75
1.40	1.60	12.9	12.21	1.03	90.1	3.85	0.79
1.60	1.90	3.8	31.77	1.49	93.9	4.98	0.82
1.90		6.1	70.94	10.78	100.00	8.99	1.42
		Sunnyside Coal					
	1.30	23.6	0.57	0.57	23.6	0.57	0.57
1.30	1.40	67.1	1.66	0.57	90.7	1.38	0.57
1.40	1.60	5.4	13.82	0.54	96.1	2.07	0.57
1.60	1.90	0.8	38.26	0.48	96.9	2.37	0.57
1.90		3.1	81.10	0.67	100.0	4.85	0.57

As expected, the ash contents were relatively similar for the three coals for the same incremental specific gravity fraction. However; since the weight distribution among the fractions varied between the coals, the ash content of the cumulative fractions also varied somewhat between the coals.

The curves in Figure 1 indicate that the recoveries were similar for the Indiana No. 3 and Sunnyside coals. This suggests comparable coal-mineral association for these coals, provided that their mineralogical properties are similar. The recovery was notably lower for the Upper Freeport coal. Much more sample was found in the 1.4-1.9 specific gravity range (as seen from Table 2). This resulted in a higher ash content for the 1.6 and 1.9 sp. gr. float product.

Results of the froth flotation tests are shown in Table 3 and Figure 2. The recovery curves for all three coals flattened out after about 6 minutes collection time. The Upper Freeport coal appears to have been more responsive to the froth flotation, showing more recovery at shorter times. However, this reflects more the hydrophobicity of the coal than the degree of association of the mineral matter with the coal.

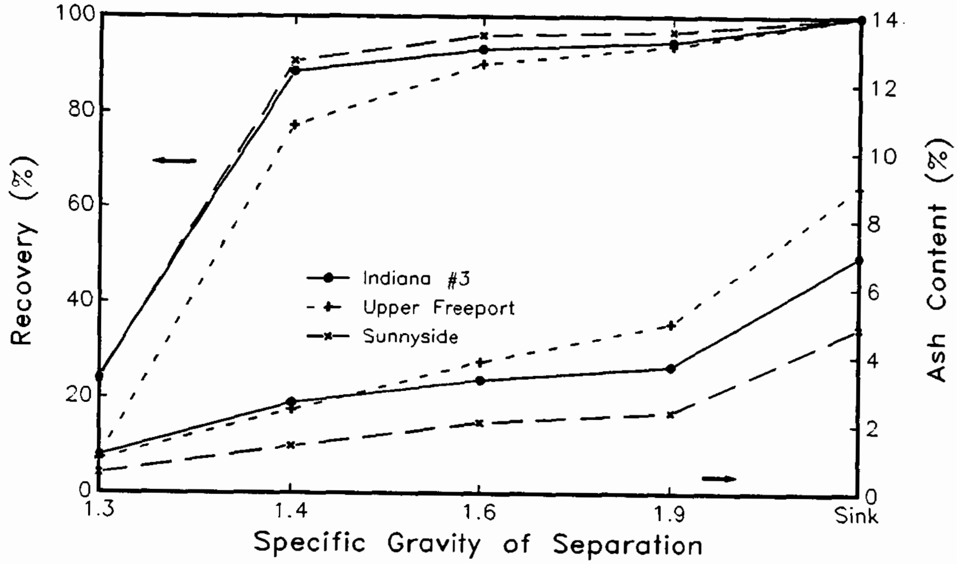

Figure 1. Recovery and ash content as a function of the specific gravity of float-sink separations

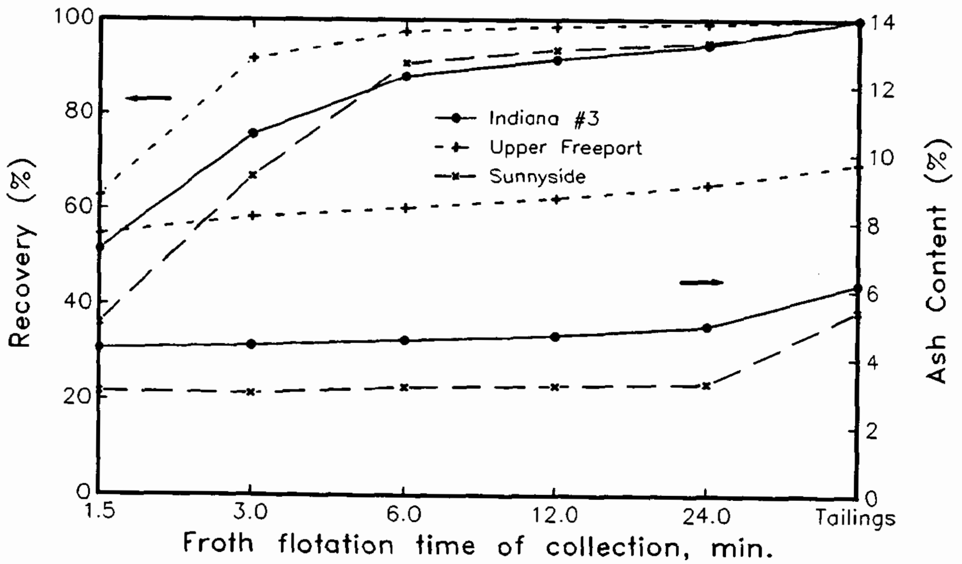

Figure 2. Recovery and ash content as a function of flotation time

Table 3. Froth flotation results for all three coals using 28 ppm MIBC and differing ml of kerosene per kg of coal (results are expressed as % of dry coal)

Flotation Time, Total Minutes	Incremental Float Product			Cumulative Float Product		
	Weight	Ash	S(tot)	Weight	Ash	S(tot)
	Indiana No. 3 coal(using 1.92 ml kerosene)					
1.5	51.5	4.30	3.34	51.5	4.30	3.34
3.0	24.2	4.58	3.26	75.7	4.39	3.31
6.0	12.3	5.53	3.49	88.0	4.55	3.34
12.0	3.6	8.27	4.04	91.6	4.69	3.37
24.0	3.0	13.23	5.06	94.6	4.97	3.42
Tailings	5.4	27.62	8.78	100.0	6.19	3.71
	Upper Freeport coal (using 0.54 ml kerosene)					
1.5	62.8	7.66	1.36	62.8	7.66	1.36
3.0	28.9	9.29	1.53	91.7	8.17	1.41
6.0	5.9	12.59	1.83	97.6	8.44	1.44
12.0	1.0	36.25	4.27	98.6	8.73	1.47
24.0	0.6	77.86	8.46	99.2	9.12	1.51
Tailings	0.8	85.65	9.17	100.0	9.74	1.57
	Sunnyside coal (using 0.55 ml kerosene)					
1.5	36.0	3.03	0.56	36.0	3.03	0.56
3.0	30.9	2.95	0.58	66.9	2.99	0.57
6.0	23.9	3.67	0.57	90.8	3.17	0.57
12.0	2.8	4.59	0.59	93.6	3.21	0.57
24.0	1.5	7.37	0.58	95.1	3.28	0.57
Tailings	4.9	46.93	0.63	100.0	5.41	0.57

From Figures 1 and 2, it is also evident that much less ash reduction was achieved by froth flotation than by float-sink separation for all three coals. Since froth flotation is a surface-sensitive process, small amounts of coal attached to mineral particles can result in these mineral particles being carried along to the froth. This effect appears to be especially significant for the Upper Freeport coal, for which minimal ash reduction was observed, apparently due to its great hydrophobicity. Examination of ash content of the incremental fractions in Table 3 indicates that after 6 minutes, the ash content of the incrementally recovered material rises dramatically, nullifying the small amount of ash reduction achieved previously. For the Upper Freeport coal, practically all of the mineral matter could float if given enough time. For the Indiana No. 3 and Sunnyside coals, on the other hand, some mineral matter simply would not float at all.

AIA results compared to float-sink results

The association results for the three coals, included in Table 4, show the incremental and cumulative percent distributions of coal, mineral matter, and coal+mineral matter as a function of the amount of mineral matter apparent in the particle cross sections. The cumulative distribution relates how much of the original sample (i.e., coal plus mineral matter) could be recovered by collecting particles of a certain mineral matter content.

The mineral matter content in a particle, listed in column 1 of Table 4, can be translated to a specific gravity estimate, assuming a coal density of 1.30 and an average mineral density of 2.60. Thus, a specific gravity of 1.3 corresponds to 0% mineral matter, 1.4 to 20%, 1.6 to 40%, 1.9 to 60%, and a specific gravity of more than 1.9 corresponds to 100%. In this manner, the distributions of Table 4 may be correlated with float-sink results. Such AIA results are plotted in Figure 3, and they can be used for comparison with the float-sink separation results in Figure 1.

Several parallel trends may be noted between the AIA and float-sink results in Figures 3 and 1, respectively. The recovery curves for the three coals generally appear in the same order in both figures. The recovery of the Sunnyside coal is greater than that of the other two coals, and the Indiana No. 3 coal generally shows higher recovery than the Upper Freeport coal for separations at specific gravities greater than 1.4. Likewise, the ash (or mineral) content curve for the Sunnyside coal is consistently below those of the other two coals. Unfortunately, the influence of stereological effects on the AIA results prevents a completely quantitative comparison of the AIA and float-sink results.

However, because the same qualitative trends appear in both figures, AIA results can be more useful since they can be obtained much faster than the float-sink results. During routine operation, sample preparation for AIA requires about 2 hours and the analysis less than 20 hours per sample. At least one sample per day can be analyzed with our equipment, but allowance for sample preparation and data reduction could result in a two-day sample turnaround. Such throughput is better that what can be achieved by testing with float-sink separation followed by ash analysis.

AIA results compared to froth flotation results

It is more difficult to relate the AIA results to froth flotation results. No comparable set of curves can be produced using the AIA results as they appear in Table 4. However, some useful information can still be obtained. The AIA results in Table 4 do show more association of minerals with the Upper Freeport coal. The plots in Figure 4 show the distribution of coal and mineral matter for the mixed (i.e., coal plus mineral matter) particles of the three coals taken from Table 4; free coal has been excluded. It can now be seen graphically that the Upper Freeport coal contains more mineral matter in this type of particle. In fact, the average mineral content of the mixed particles (i.e., the associated coal-mineral fraction) of the Upper Freeport coal is 45%, compared to only 26% and 29% for the Indiana No. 3 and Sunnyside coals, respectively.

However, even with these coal-mineral association insights, the previously stated tendency for the Upper Freeport coal to respond more to froth flotation than the other two coals appears to be the determining factor in the poor mineral removal for this coal. Apparently, the mineral matter in the Upper Freeport coal can float, even with very little coal associated with it. However, this issue cannot be answered by AIA alone and requires further study by other analytical techniques.

Table 4. Distributions (in %, dry basis) of coal and mineral matter for all three coals, as a function of particle mineral matter (MM) content

MM Content	Incremental			Cumulative			% MM in Cumulative Total
	Coal	MM	Total	Coal	MM	Total	
Indiana No. 3 coal							
0	61.61	0.00	61.61	61.61	0.00	61.61	0.00
0-10	14.32	0.58	14.90	75.93	0.58	76.50	0.75
10-20	5.51	1.04	6.55	81.44	1.61	83.05	1.94
20-30	2.86	0.96	3.82	84.30	2.57	86.88	2.96
30-40	2.34	1.27	3.61	86.64	3.85	90.49	4.25
40-50	0.87	0.72	1.59	87.51	4.57	92.08	4.96
50-60	0.96	1.17	2.13	88.47	5.73	94.21	6.09
60-70	0.65	1.27	1.92	89.12	7.00	96.12	7.28
70-80	0.53	1.62	2.15	89.65	8.62	98.28	8.78
80-90	0.20	1.14	1.34	89.85	9.77	99.62	9.80
90-100	0.01	0.36	0.38	89.87	10.13	100.00	10.13
Total	89.87	10.13	100.00				
Upper Freeport coal							
0	73.35	0.00	73.35	73.35	0.00	73.35	0.00
0-10	4.62	0.20	4.82	77.97	0.20	78.17	0.26
10-20	3.32	0.57	3.88	81.28	0.77	82.06	0.94
20-30	1.25	0.43	1.68	82.53	1.20	83.73	1.43
30-40	1.24	0.64	1.88	83.77	1.84	85.61	2.15
40-50	0.95	0.75	1.70	84.72	2.59	87.32	2.97
50-60	1.14	1.30	2.44	85.86	3.89	89.75	4.34
60-70	0.94	1.79	2.73	86.81	5.68	92.48	6.14
70-80	0.31	0.88	1.19	87.11	6.56	93.68	7.01
80-90	0.67	3.83	4.50	87.78	10.39	98.17	10.59
90-100	0.11	1.71	1.82	87.89	12.11	100.00	12.11
Total	87.89	12.11	100.00				
Sunnyside coal							
0	84.28	0.00	84.28	84.28	0.00	84.28	0.00
0-10	6.06	0.32	6.38	90.34	0.32	90.66	0.35
10-20	1.88	0.31	2.19	92.22	0.63	92.85	0.68
20-30	0.86	0.27	1.13	93.08	0.90	93.97	0.95
30-40	0.66	0.37	1.04	93.74	1.27	95.01	1.34
40-50	0.85	0.71	1.56	94.59	1.98	96.57	2.05
50-60	0.27	0.32	0.59	94.86	2.30	97.16	2.37
60-70	0.25	0.46	0.71	95.11	2.77	97.88	2.83
70-80	0.25	0.74	0.99	95.36	3.51	98.87	3.55
80-90	0.10	0.55	0.65	95.46	4.06	99.52	4.08
90-100	0.04	0.44	0.48	95.49	4.51	100.00	4.51
Total	95.49	4.51	100.00				

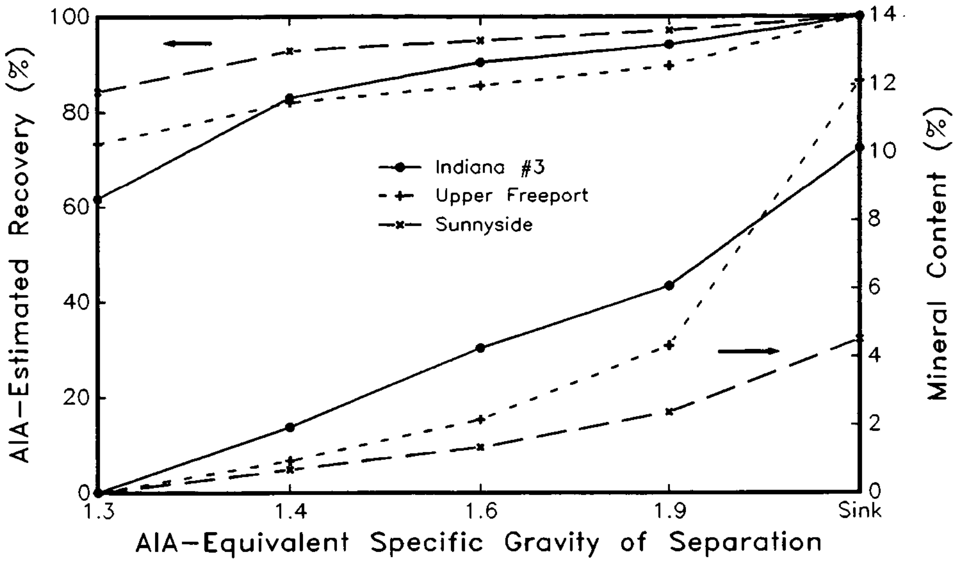

Figure 3. AIA-estimated recovery and mineral content as a function of AIA-equivalent specific gravity

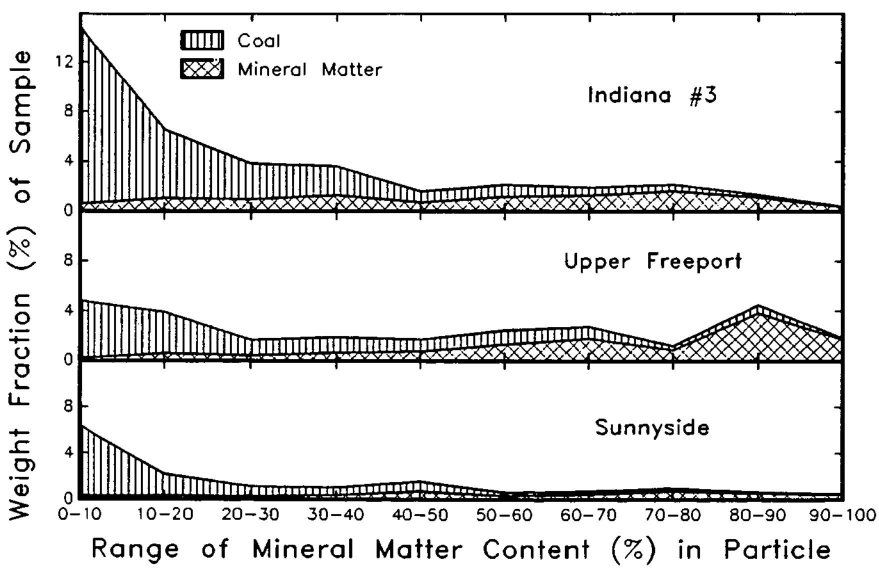

Figure 4. Distribution of coal and mineral matter as a function of weight fraction of mineral matter in the particles

CONCLUSIONS

Automated image analysis was used to determine the association of mineral features with coal, and the results were expressed in a format that provided insight into the float-sink response of the sample. Qualitatively similar trends were observed for the conventional float-sink and the AIA results; however, quantitative comparisons are currently limited by stereological effects on the AIA results.

The AIA results, as developed and presented in this work, provide less direct insight into the froth flotation behavior. However, there did appear to be a correlation between the large amount of mineral matter in the mixed particles of the associated coal and mineral fraction measured by AIA and the poor ash rejection from the Upper Freeport coal during froth flotation. Work is continuing on other AIA reporting formats which could be more directly related to froth flotation.

AIA results can be obtained considerably quicker than results from either of the conventional cleaning tests. This suggests that AIA may be useful in monitoring the character of feed to a preparation plant, particularly since AIA is able to detect differences in the association of coal and mineral matter particles.

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